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From: Salemi, Charles (DPH)
Sent: Monday, April 25, 2011 11:25 AM
To: Nassif, Julianne (DPH); Lawler, Michael (DPH); Piro, Peter (DPH)
Subject: RE: 1,4-BD Retraction

Hi all, I thought 1,4-Butanediol could be seen on the GC or GCMS without any cleanup? Just dilute it and shoot. What did I miss here? CBS

From: Nassif, Julianne (DPH)
Sent: Monday, April 25, 2011 10:32 AM
To: Lawler, Michael (DPH); Piro, Peter (DPH); Salemi, Charles (DPH)
Subject: RE: 1,4-BD Retraction

Talk about timing…I spent a good portion of the weekend with this article (ML see me if you need a copy). Ethyl acetate was indeed determined to be the best solvent (although later Wang determine a c80:20 mix of CHCl3 and n-butanol had even better efficiency). The polar functional groups, especially the oxygen containing ones are the key.

Other key factors are pH (the pKa is 4.6) so you need to be between pH 2-4, ionic strength (5 M NaCl) and if you want to eliminate GBL pre-wash w/ chloroform to remove the neutrals.

Julie

PS time is also a factor as the stability of the free acid is not long term

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From: Lawler, Michael (DPH)
Sent: Monday, April 25, 2011 8:10 AM
To: Piro, Peter (DPH); Salemi, Charles (DPH)
Cc: Nassif, Julianne (DPH)
Subject: RE: 1,4-BD Retraction

Peter,
I am redrafting again the GHB screen protocol from the front end of the analysis. With that in mind and in consideration of the remaining levels of samples to prep and use, could you give me copies of the literature which supports this? I'd like to review the material before I do another prep, particularly in light of the apparent failure of the New Hampshire State Police scheme we attempted most recently.
Thanks, Mike

From: Piro, Peter (DPH)
Sent: Monday, April 25, 2011 7:52 AM
To: Lawler, Michael (DPH); Salemi, Charles (DPH)
Cc: Nassif, Julianne (DPH)
Subject: 1,4-BD Retraction

Hello All,
To extract 1,4-BD from an aqueous sample, I'm suggesting we try a method similar to the extraction of GHB free acid. Aqueous samples are first saturated with NaCl and then extracted 2X with ethyl acetate in a 1:2 ratio (water:ethyl acetate). Excess ethyl acetate is then evaporated while making sure not to evaporate to dryness. During my last round of experiments with 1,4-BD I must have

isolated the wrong layer, i.e. the H2O layer instead of the petroleum ether layer….mi culpa. It turns out that petroleum ether, chloroform and ethyl ether have limited success with BD extractions. Ethyl acetate seems to be a better extracting solvent and the trickery with NaCl is beneficial for total recovery levels.